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Drift Monitors - Drift Correction for XRF



Drift correction for XRF

XRF is characterized by a very high long-term stability. Nevertheless, the components show certain wear effects over time so that the measured signal changes. If the change is known, it can be corrected for using a so-called drift correction.

The drift correction is based on the principle that at the time of the calibration (day 0) and at a given time afterwards (day 1), the same, stable drift correction sample is measured. The correction is then calculated from the change in the measured signal.

Requirements for a Drift Correction Sample:

- High signal stability
- High intensity
- Same elements as in the application
- Easy cleaning
- Replaceability



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Terminology often used for these samples:

- Drift correction samples
- Drift monitor
- Monitor samples
- Setting up samples (SUS)
- Monitor standards
- Recalibration sample

To use the drift correction, the instrument software must make a connection between the measurement of a line on the drift correction sample and the measurement of a line in a measuring method.

This takes for granted that the measuring conditions are identical or similar. It also assumes that measurement of the drift correction sample is subject to the same changes as measurement in a measuring method.

At least one million counts (measurement error < 0.1%) should be recorded for the main components in order to keep the measurement error as small as possible.

Requirements for the Measuring Conditions:

- Measurement error must be as small as possible
- Measuring parameters must be consistent with the test method
- Different measuring parameters require their own drift correction

Fundamentally, There Are Two Procedures for Drift Correction:

- Based on a high sample (high element concentration)
- Based on a high and low sample (low element concentration)

Figure 1 shows the drift correction based on a high sample. An additional background measurement must be conducted to include changes in the background during trace analysis.



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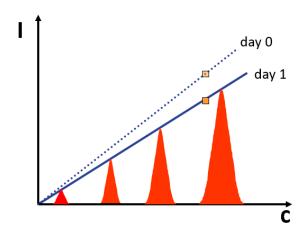


Fig. 1 Drift correction based on a high sample. For trace analysis the additional measurement of background is obligatory.

In contrast, Figure 2 shows drift correction based on a high and a low sample. Here it is not necessary to conduct a background measurement, which is why this is used for simultaneous spectrometers. It is necessary to be sure that the intensity of the low sample is as close to the lowest calibration sample as possible. In addition, the intensities of the high and low samples for an element must be different by at least one order of magnitude. This prevents overcorrection by excessive extrapolation.

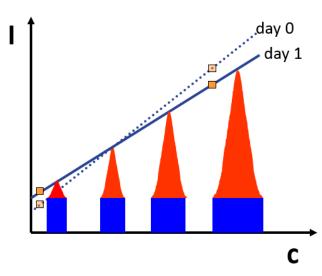


Fig. 2 Drift correction based on a high and low sample. No additional background measurement necessary.



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Selection of Drift correction samples

To make drift monitoring and correction simple FLUXANA has developed a universal drift monitor set FLX-DC. It consists of 6 samples which cover nearly the whole periodic table. Fig. 4 gives an indication which elements are covered as low and high sample. With this set you will be able to establish drift monitoring for 99% of all common applications.



Fig. 3 Universal drift monitor set FLX-DC consisting of 6 samples FLX-DC 01 to FLX-DC 06.

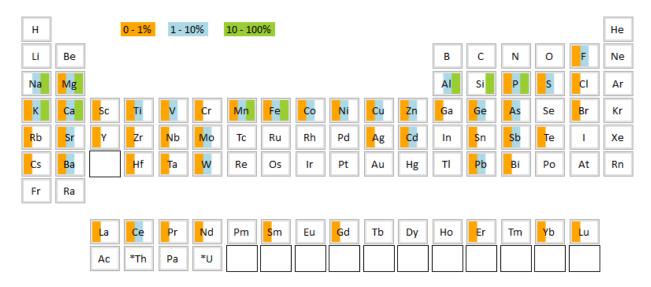


Fig. 4 Indication of concentrations covered by the set FLX-DC.

There is a second version of the DC Set with additional elements * Th and U.



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An extension of the set was created for low SiO2 concentrations. As most glasses are based on SiO2, it is sometimes difficult to find glasses with low SiO2 concentrations. FLUXANA has therefore produced a set of phosphate glasses that do not require SiO2 as a glass former. In addition to a blank glass, 2 glasses were produced with 1% and 5% SiO2 doping.

Long Term Stability of Drift correction samples

The following diagrams show a typical monitoring of a fluorescence line with atomic number Z >= 19 on a wavelength dispersive spectrometer using a FLUXANA drift monitor. The data are uncorrected, i.e. they show the raw intensity from the instrument over a period of 20 months. The relative standard deviation calculated for that period is 0.5%. This includes the total measurement error, all observed instrument instabilities and any instability of the drift monitor sample.

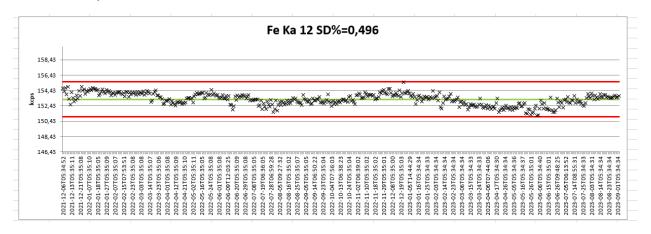


Fig. 5 Drift of Fe Ka Line within 2 years

If we investigate a light element with atomic number Z < 19 we see already a bigger decrease in signal over the same period. Reason for that is that the signal is influenced stronger by critical parts of the instrument like vacuum, humidity, absorption layers, dust, etc. The relative standard deviation calculated for that special line P Ka is around 1%.



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P Ka 12 SD%=1,043

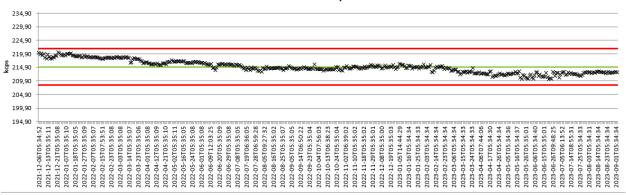


Fig. 6 Drift of P Ka Line within 2 years



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Example: Drift of a 4kW WDXRF instrument in 5 years

The following figure shows the raw intensity measured at the same FLUXANA drift monitor within 5 years. There could be 2 groups identified the one around 5% intensity loss and the one > 10% intensity loss. The second group with > 10% intensity loss represents fluorescence lines with very low fluorescence yield and low energy which are more sensitive to vacuum, absorption layers on tube or detector window or contaminations generally.

Within the first group with an intensity loss around 5% we could also see that the loss is very consistent over many different element lines. This could be seen as prove to the long-term stability of the FLUXANA drift monitor glasses which means there is no movement of elements inside the sample or to the sample surface. Otherwise, we would see a much higher spread in intensity changes over the long time.

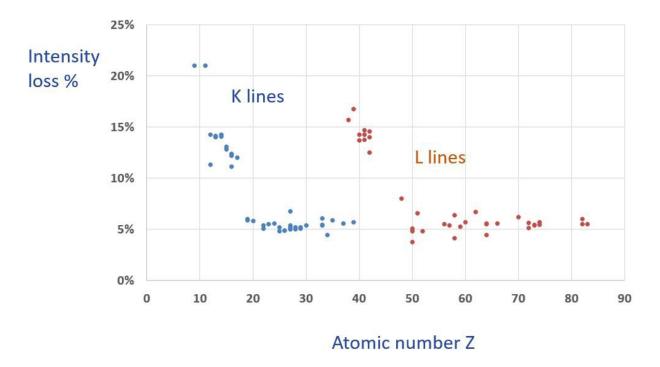


Fig. 7 Observed drift/intensity loss of K and L lines at a WDXRF instrument within 5 years



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Commercially available sets of drift monitors

FLX-DC Universal set FLX-DC01, FLX-DC02, FLX-DC03, FLX-DC04, FLX-DC05, FLX-DC06

FLX-DC-UT Universal set with U and Th, FLX-DC02, FLX-DC03, FLX-DC04, FLX-DC05, FLX-DC06, FLX-DC0

DC07

FLX-SiO2 Extension drift monitor set with low SiO2 concentrations, FLX-SiO2-00 0% SiO2,

FLX-SiO2-01 1% SiO2 and FLX-SiO2-05 5% SiO2

Literature

[1] Rainer Schramm, Xay Fluorescence Analysis: Practical and Easy, 2nd edition, FLUXANA (2017)

[2] www.fluxana.com